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मानक

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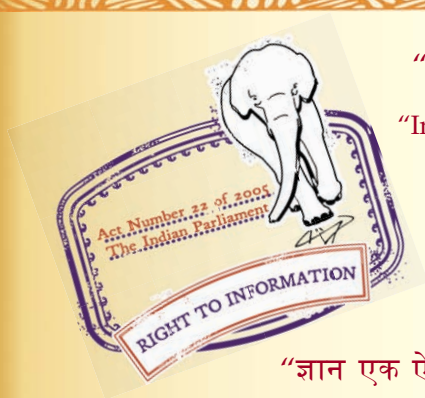
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“Step Out From the Old to the New”

IS 7362 (1986): Tobias Acid [PCD 9: Organic Chemicals  
Alcohols and Allied Products and Dye Intermediates]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”



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IS : 7362 - 1986

*Indian Standard*  
SPECIFICATION FOR  
TOBIAS ACID  
( *First Revision* )

UDC 667.281 : 547.654.1/.2



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**INDIAN STANDARDS INSTITUTION**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

**AMENDMENT NO. 1 MARCH 2002**  
**TO**  
**IS 7362 : 1986 SPECIFICATION FOR TOBIAS ACID**  
**( First Revision )**

( *Page 3, clause 0.2, Structural Formula* )— Insert the following below the structural formula:

‘( CAS No. 81-16-3 )’.

[ *Page 4, Table 1, Sl No.(iii)* ] — **Substitute the following for the existing:**

(1)	(2)	(3)	(4)
iii)	2-Napthylamine content, ppm, <i>Max</i>	100	A-4

( PCD 11 )

Reprography Unit, BIS, New Delhi, India

# *Indian Standard*

## SPECIFICATION FOR TOBIAS ACID

### ( *First Revision* )

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( *Continued on page 2* )

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**IS : 7362 - 1986**

( Continued from page 1 )

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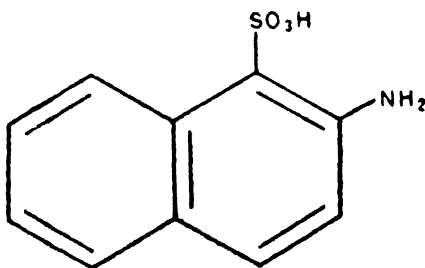
***Indian Standard***  
**SPECIFICATION FOR**  
**TOBIAS ACID**  
**( *First Revision* )**

## 0. FOREWORD

**0.1** This Indian Standard ( First Revision ) was adopted by the Indian Standards Institution on 26 February 1986, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

**0.2** This standard was first issued in the year 1974. The Committee responsible for the preparation of this standard decided to revise it in order to update the requirement of assay and matter insoluble in dilute sodium hydroxide solution in accordance with the development in the industry over the last decade.

**0.3** Tobias acid (  $C_{10}H_9O_3NS$  ) also described by such chemical names as 2-naphthylamine-1-sulphonic acid and 2-amino-1-naphthalene sulphonic acid is an important dye intermediate used mainly in production of red yellow organic lakes and naphthol AS-SW. It is represented by the following structural formula:



TOBIAS ACID  
 2-naphthylamine-1-sulphonic acid  
 ( Molecular mass 223 )

## IS : 7362 - 1986

**0.4** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960\*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

### 1. SCOPE

**1.1** This standard prescribes the requirements and the methods of sampling and tests for tobias acid.

### 2. REQUIREMENT

**2.1 Description** — The material shall be in the form of a paste or, if dry, in the form of pinkish crystalline powder containing a few soft lumps.

**2.2** The material shall also comply with the requirements given in Table 1.

**TABLE 1 REQUIREMENTS FOR TOBIAS ACID**

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO CL No. IN APPENDIX A
(1)	(2)	(3)	(4)
i)	Assay ( as total amine ), percent by mass ( on dry basis ), <i>Min</i>	98.5	A-2
ii)	Insolubles in dilute sodium hydroxide solution, percent by mass ( on dry basis ), <i>Max</i>	0.2	A-3
iii)	2-Naphthylamine content, percent by mass, <i>Max</i>	0.5	A-4

### 3. PACKING AND MARKING

**3.1 Packing** — The material shall be packed in steel drums ( see IS : 2552-1979† ) lined with suitable polyethylene film, or as agreed to between the purchaser and the supplier.

\*Rules for rounding off numerical values ( *revised* ).

†Specification for steel drums ( galvanized and ungalvanized ) ( *second revision* ).

**3.2 Marking** — Each container shall be securely closed and shall bear legibly and indelibly the following information:

- a) Name of the material;
- b) Name of the manufacturer and his recognized trade-mark, if any;
- c) Batch number;
- d) Gross, net and tare mass; and
- e) The minimum cautionary notice worded as under:

‘IT IS A MILD SENSITIZER. LOCAL CONTACT MAY CAUSE DERMATITIS.’

**3.2.1** The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution ( Certification Marks ) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

#### 4. SAMPLING

**4.1** The material shall be sampled in accordance with 3 of IS : 5299-1969\*.

#### 4.2 Number of Tests

**4.2.1** Test for assay shall be conducted on each of the individual samples separately.

**4.2.2** Tests for the determination of the remaining characteristics, namely, insolubles in dilute sodium hydroxide solution and 2-naphthylamine content, shall be conducted on the composite sample.

#### 4.3 Criteria for Conformity

**4.3.1 For Individual Samples** — The lot shall be declared as conforming to the requirement of assay, if each of the individual test results satisfies the relevant requirement given in Table 1.

**4.3.2 For Composite Sample** — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample ( see 4.2.2 ), the test result for each of the characteristics shall satisfy the relevant requirement given in Table 1.

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\*Methods of sampling and tests for dye intermediates.

**4.3.3** The lot shall be deemed to conform to the requirements of the specification, if conditions laid down in **4.3.1** and **4.3.2** are satisfied.

## **5. TEST METHODS**

**5.1** Tests shall be carried out according to the methods prescribed in Appendix A, as indicated in col 4 of Table 1.

**5.2 Quality of Reagents** — Unless specified otherwise, pure chemicals and distilled water ( *see* IS : 1070-1977\* ) shall be employed in tests.

NOTE - 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

# **A P P E N D I X A**

( *Table 1* )

## **METHODS OF TESTS FOR TOBIAS ACID**

### **A-1. PREPARED SAMPLE**

**A-1.1** If the sample is dry, mix well by rotating the bottle several times. If the sample is in the form of paste, mix well. Dry the material at  $105 \pm 2^{\circ}\text{C}$  to constant mass. Use this *prepared sample* for tests.

### **A-2. ASSAY**

**A-2.0 Outline of the Method** — The material is dissolved in aqueous sodium hydroxide. A known volume of the solution is titrated in acidic medium with standard sodium nitrite solution, using starch-iodide test paper as an external indicator.

#### **A-2.1 Reagents**

**A-2.1.1 Sodium Hydroxide Solution** — approximately 10 percent ( *m/v* ).

**A-2.1.2 Concentrated Hydrochloric Solution** -- *see* IS : 265-1976†.

**A-2.1.3 Standard Sodium Nitrite Solution** -- 0.1 N.

**A-2.1.4 Starch-iodide Test Papers**

#### **A-2.2 Procedure**

**A-2.2.1** Weigh accurately about 11 g of the *prepared sample* ( *see* **A-1.1** ) and dissolve with the requisite amount of sodium hydroxide solution and about 300 ml of water. Heat, if necessary. Cool to room temperature and make up to volume with water, in a 500-ml volumetric flask. Mix well.

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\*Specification for water for general laboratory use ( *second revision* ).

†Specification for hydrochloric acid ( *second revision* ).

**A-2.2.2** Pipette 50 ml aliquot of this solution in a one-litre beaker and dilute with cold water to about 600 ml. Then add 35 ml of hydrochloric acid. Cool to 15 to 20°C and titrate, while stirring mechanically, with standard sodium nitrite solution which is added through a thistle funnel, the end of which is well under the solution. Add the sodium nitrite solution as rapidly as it is consumed. The end point is reached when a drop of the solution spotted on a starch-iodide test paper produces an immediate blue-coloured ring which may be repeatedly obtained during next 5 minutes without further addition of sodium nitrite.

NOTE 1 — Precipitation during diazotization, if observed, may be avoided by further dilution.

NOTE 2 — Temperature during diazotization shall be between 15 to 20°C.

### A-2.3 Calculation

$$\text{Assay, percent by mass (Molecular mass : 223)} = \frac{V \times N \times 223}{M}$$

where

$V$  = volume in ml of the standard sodium nitrite solution used;

$N$  = normality of sodium nitrite solution; and

$M$  = mass in g of the sample taken for the test.

## A-3. DETERMINATION OF INSOLUBLES IN SODIUM HYDROXIDE SOLUTION

**A-3.1 Procedure** — Weigh accurately about 10 g of the *prepared sample* (see A-1.1). Transfer to a 500-ml beaker. Add 300 ml of water and sodium hydroxide solution slowly (10 percent  $m/v$ ) while stirring with a rod until distinctly alkaline to phenolphthalein test paper. Stir until dissolved. Heat, if necessary, to dissolve completely. Filter through a tared sintered or Gooch crucible; wash with hot water till filtrate is free from alkali. Dry in an oven at  $100 \pm 2^\circ\text{C}$ . Cool in a desiccator and weigh. Calculate percentage residue.

## A-4. DETERMINATION OF 2-NAPHTHYLAMINE CONTENT

### A-4.1 Method I (Titrimetric)

**A-4.1.1 Outline of the Method** — The sample is dissolved in aqueous sodium hydroxide. The solution is extracted with ether when free 2-naphthylamine dissolves in ether layer. The amine in the ether layer is then estimated.

#### A-4.1.2 Reagents

**A-4.1.2.1 Sodium hydroxide solution** — approximately 10 percent ( $m/v$ ).

**A-4.1.2.2 Hydrochloric acid** — 1 : 1 ( *v/v* ).

**A-4.1.2.3 Standard sodium nitrite solution** — 0.1 N.

**A-4.1.2.4 Starch-iodide test paper**

**A-4.1.2.5 Ether** — reagent grade.

**A-4.1.3 Procedure** — Weigh accurately about 10 g of the *prepared sample* ( see **A-1.1** ) into a 500-ml beaker. Add 200 ml of water and sufficient quantity of sodium hydroxide solution until the solution is alkaline to brilliant yellow paper and the tobias acid sample completely goes in solution. Transfer the solution into a 500-ml separating funnel. Add 50 ml of ether and shake well. Care shall be taken to see that the pressure developed inside the funnel is released while shaking. Allow to separate and draw out the ether layer. Repeat this extraction twice over and combine all the ether layers. Wash the combined ether extract with 100 ml of water. Take the ether extract in a beaker. Add 100 ml of hydrochloric acid and keep the beaker in a hot water-bath. When all the ether has evaporated, titrate the hydrochloric acid solution against standard sodium nitrite solution as prescribed in **A-2.2.2**.

**A-4.1.4 Calculation**

$$\text{Free naphthylamine content, percent by mass} = \frac{V \times N \times 14.3}{M}$$

where

*V* = volume in ml of sodium nitrite solution consumed,

*N* = normality of sodium nitrite solution, and

*M* = mass in g of the sample taken for the test.

**A-4.2 Method II ( Thin Layer Chromatography )**

**A-4.2.1 Reagents**

**A-4.2.1.1 Tobias acid** — 2-naphthylamine-free.

**A-4.2.1.2 2-naphthylamine** — pure.

**A-4.2.1.3 Benzene** — reagent grade.

**A-4.2.1.4 Methanol** — reagent grade.

**A-4.2.1.5 Liquor ammonia** -- reagent grade.

**A-4.2.1.6 Spray reagent** — Dissolve 0.1 percent diazo fast red B in water. Filter and use as spray reagent.

**A-4.2.2 Apparatus**

**A-4.2.2.1 Thin layer glass plate** —  $10 \times 20$  cm, coated with silica gel G in a thickness of 250 micron and activated at  $110^{\circ}\text{C}$  for 20 minutes.

**A-4.2.2.2 Micro pipette** — 10 microlitre capacity.

**A-4.2.2.3 Developing chamber** — Thin layer chromatographic rectangular glass developing chamber which contains the eluent benzene + methanol (98 : 2) (v/v) and which is closed well with lid for saturation.

**A-4.2.2.4 Chromatographic sprayer**

**A-4.2.3 Procedure** — Prepare standard solution of tobias acid containing known amount of 2-naphthylamine in the following way:

Into each of three 100-ml standard volumetric flasks, weigh accurately 2.5 g of the tobias acid. Then add 1.5 ml, 2 ml and 2.5 ml of a 0.5 percent solution of 2-naphthylamine in methanol to flask No. 1, No. 2 and No. 3 respectively. Dissolve the contents of the flask by adding 50-ml of methanol and 5 ml of liquor ammonia. Then make the volume up to mark with methanol. Thus there are three solutions of 0.3 percent, 0.4 percent and 0.5 percent 2-naphthylamine content.

In the fourth flask, weigh accurately 2.5 g of the *prepared sample* (see A-1.1) under test and make up the volume by first dissolving in 10 ml methanol and 5 ml of liquor ammonia and then finally with methanol to 100 ml.

Place 10 microlitre spot of each of the four solutions, using micro-pipette in the same line at a distance of about 1.5 to 2.0 cm from the bottom. Allow to dry. Then place the plate in the solvent of the chamber. Allow the solvent to run about 15 cm. Take out the plate after 15 cm run and dry the solvent completely. Then spray the plate, with spray reagent. Heat the plate in an air oven at about  $50^{\circ}\text{C}$  for about 10 minutes. Take out the plate from the oven and observe the separated spots characteristic of constituents identifiable by their colour as under:

Constituents	Zone	Rf-Value	Colour
Tobias acid	I	0.00	Orange
2-Naphthylamine	II	0.77	Violet

**A-4.3 Report** — Compare the intensity of the spots visually with those of known standard and report 2-naphthylamine content as that which is nearest in intensity to the standard. In case the colour intensity does not come in the range of the standard spots, repeat the whole procedure using different percentages of 2-naphthylamine.

## INTERNATIONAL SYSTEM OF UNITS ( SI UNITS )

### Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

### Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

### Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 kg.m/s <sup>2</sup>
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m <sup>2</sup>
Frequency	hertz	Hz	1 Hz = 1 c/s (s <sup>-1</sup> )
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m <sup>2</sup>





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